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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of

Maria Anna Wubben et al.

Serial No. **08/776,321** Examiner: **C. E. Sherrer**

Filed: **August 3, 1995** Art Unit: **1302**

For: **PECTINS AS FOAM STABILIZERS FOR BEVERAGES HAVING A FOAM HEAD**

D E C L A R A T I O N

The Assistant
Commissioner of Patents
Washington , D.C. 20231

Sir:

The undersigned, Alexandra Johanna Mathilda Wijsman, Secretaris Versteeglaan 37, 3451 XH Vleuten, the Netherlands, herewith declares as follows:

1. I am an employee of Heineken Technical Services B.V. at Zoeterwoude, the Netherlands. I am a graduate of the Agricultural University of Wageningen, with a specialization in food technology. For about two and a half years I have been involved in research on raw materials for the production of beer, such as polysaccharides;

2. I have performed some experiments in order to establish the effect of wort boiling on the foam stabilizing effect of pectins on the foam of beer. In these experiments, I have used a pectin that was extracted from waste material remaining after a CO_2 extraction of hops. Reference pilsner beer was used for the foam experiments;

3. The foam stability of pectin beer and the molecular weight distribution of the pectin preparations were determined after boiling (100°C) the pectin preparations for 0, 5, 10, 30 , 60 and 90 minutes. After heat treatment the pectin preparations were added to beer in the following concentrations: 30 mg/bottle (approximately 10 g/Hl) and 90 mg/bottle (approximately 30 g/Hl). The foam stability of the beer was determined with a Nibem meter. The molecular weight distribution of the pectin preparations was determined by high performance size-exclusion chromatography (3 g/ml). detection was performed with refractive index detection (RI), UV detection at 280 nm (A280), light scattering (M_w) and intrinsic viscosity (η);

4. Enclosed Figures 1 and 2 show that pectin is decomposed during boiling (100°C). The molecular weight and the intrinsic viscosity of the hop pectin preparations extensively decrease with an increasing boiling time of 0 to 90 minutes (Figure 1). Also, Figure 2 shows that boiling of hop pectin for 90 minutes results in a shift from high to low molecular weight material as shown by the declining peak at a retention time of 20-25 minutes which represents the high molecular weight population (Figure 2);

5. Enclosed Figures 3 and 4 show that the foam improvement of pectin substantially decreases at least 37 to 52% with increasing boiling time from 0 to 90 minutes at pectin concentrations of 10 g/Hl and 30 g/Hl, respectively;

6. The results of the above discussed experiments illustrate that pectins are decomposed during boiling. This results therein, that the foam stabilizing effect of pectins added at a late stage of the wort boiling is significantly better than that of pectins which are added prior to wort boiling.

7. The undersigned declares that all statements made herein are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements and the like so made may jeopardize the validity of the document, or application, or any patent issuing thereon.

Signed this 6 day of March, 1998

By 

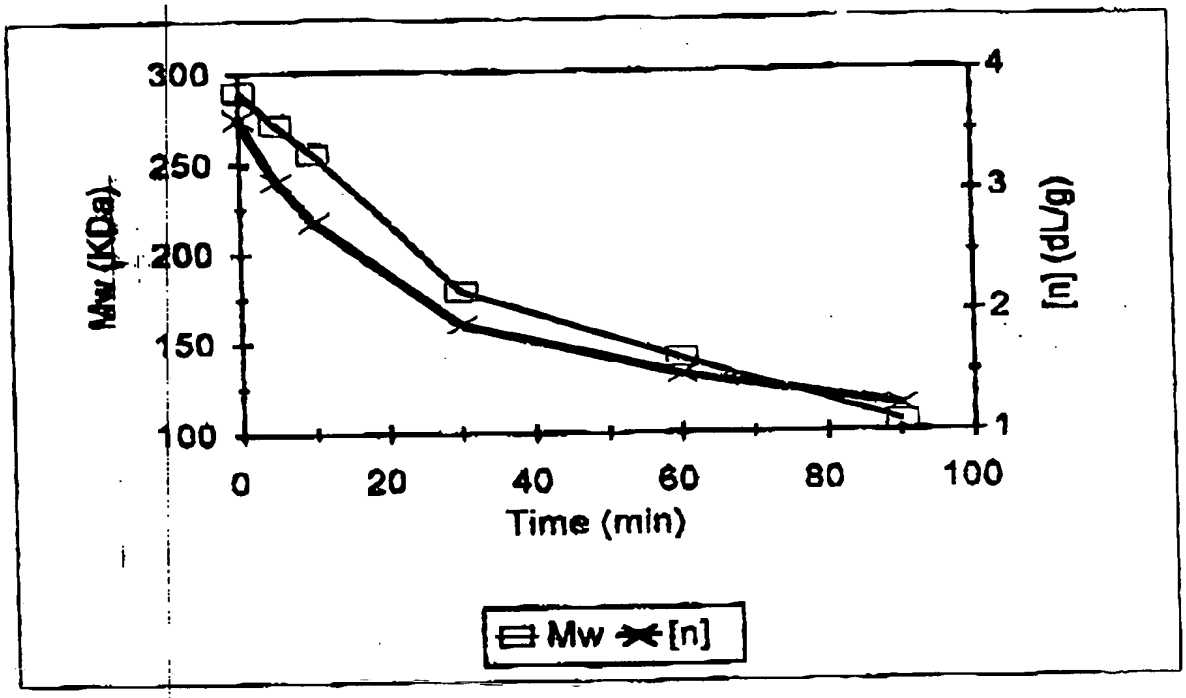

Figure 1. Development of the molecular weight (Mw) and the intrinsic viscosity (n) of hoppectin, extracted from residues of CO_2 extraction, after boiling (100°C) during 0, 5, 10, 30, 60 and 90 minutes.

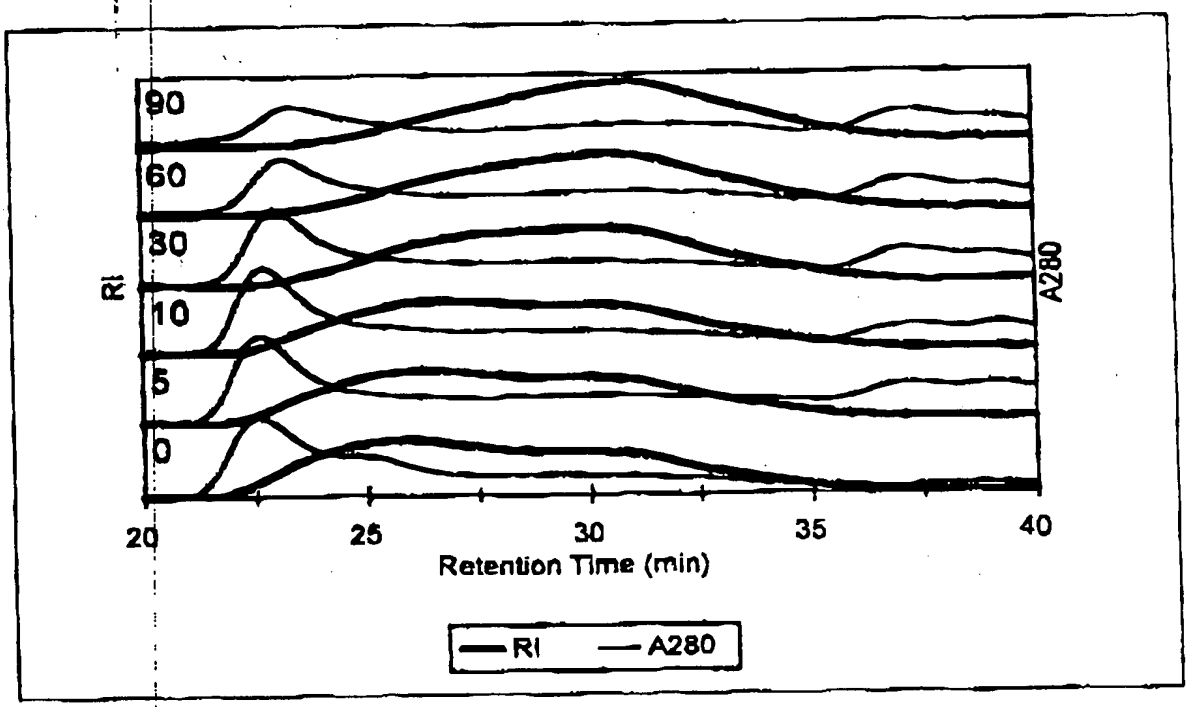

Figure 2. High-performance size-exclusion chromatography curves of hoppectin, extracted from residues of CO_2 extraction, after boiling (100°C) during 0, 5, 10, 30, 60 and 90 minutes (RI=refractive index detection; A280=UV detection at 280 nm).

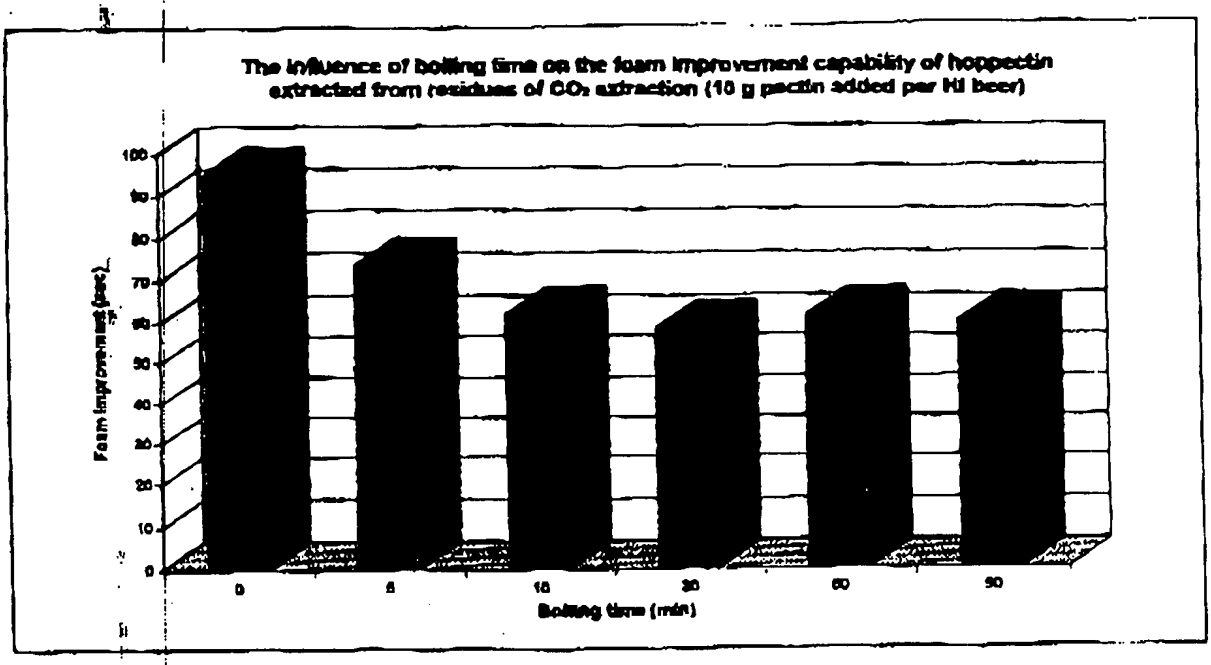

Figure 3. The foam improvement capability of beer after addition of hop pectin (30mg/bottle = approx.10g/hl), extracted from residues of CO_2 extraction, after boiling (100°C) during 0, 5, 10, 30, 60 and 90 minutes.

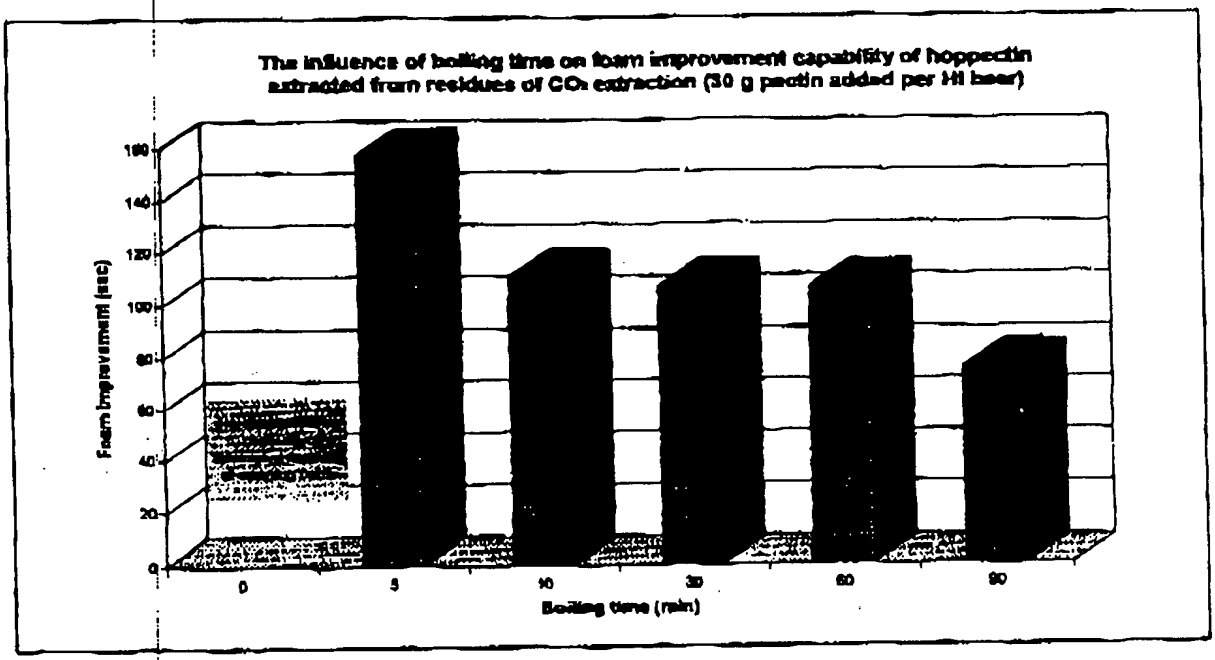

Figure 4. The foam improvement capability of beer after addition of hop pectin (90mg/bottle = approx.30g/hl), extracted from residues of CO_2 extraction, after boiling (100°C) during 0, 5, 10, 30, 60 and 90 minutes.